(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
22 November 2001 (22.11.2001)

PCT

(10) International Publication Number
WO 01/87092 A2

(51) International Patent Classification[7]: A23L 2/02, 1/068

(21) International Application Number: PCT/US01/15008

(22) International Filing Date: 1 May 2001 (01.05.2001)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
09/570,854 12 May 2000 (12.05.2000) US

(71) Applicant: **THE COCA-COLA COMPANY** [US/US]; One Coca Cola Plaza, NW, Atlanta, GA 30313 (US).

(72) Inventors: **MA, Sheng, Nue**; 1182 Brantley Estates Drive, Altamonte Springs, FL 32714 (US). **LADA, Mark, Witold**; 719 Red Wing Drive, Lake Mary, FL 32746 (US).

(74) Agents: **PAPPAS, Peter, G.** et al.; Sutherland Asbill & Brennan LLP, 999 Peachtree Street, NE, Atlanta, GA 30309-3996 (US).

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

**Published:**
*without international search report and to be republished upon receipt of that report*

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(54) Title: FRUIT JUICES HAVING ENHANCED FLAVOR AND EXTENDED QUALITY SHELF-LIFE

(57) Abstract: Fruit juices and fruit juice products having enhanced flavor and extended quality shelf-life. In particular, the present invention relates to fruit juices and fruit juice beverages that are capable of being stored in non-refrigerated conditions for extended periods of time without the development of off flavors and/or off taste. The fruit juices and fruit juice beverages comprise a juice having reduced levels of off-flavor compounds and/or reduced levels of precursors of off-flavors thereby making a juice which tastes better immediately. Additionally, since a large amount of the off flavors and/or precursors are removed, the off flavors are slowed or thereby allowing the juice to be stored under non-refrigerated conditions for one or more months without significant degradation of the flavor of the juice.

WO 01/87092 A2

# FRUIT JUICES HAVING ENHANCED FLAVOR AND EXTENDED QUALITY SHELF-LIFE

## FIELD OF THE INVENTION

The present invention is directed to fruit juices having enhanced quality, flavor, and taste. The present invention is additionally directed to the preparation of beverages, such as fruit juice beverages, to inhibit and/or reduce their tendency to deteriorate in flavor and quality during storage. The fruit juices of the present invention may be used in the preparation of whole juices, blended juices, concentrated juices or juices used as ingredients in diluted juice beverages, jams, jellies, fruit preserves or other foods that may deteriorate due to quality deterioration of fruit juice.

## BACKGROUND OF THE INVENTION

The production of beverages has grown increasingly complex. Today's consumers drink a widening array of beverages, flavors and formulations. Many of these consumers are consuming fruit juices for taste and nutritional reasons.

Fruits for juice production are generally harvested during a relatively short season each year. The juice must be prepared and then preserved until the next harvest season if it is to be available for consumption year around. This is especially true of juices from fruits that are non-climacteric. These fruits cannot be stored as fruit and have a relatively short storage life. This is due to the fact that these fruits have to be harvested in a fully mature state, which exposes them to damage and decline in the post harvest period. Fruit that is processed can be preserved for consumption by a number of methods. each having its own benefits and drawbacks.

The earliest modern method of commercially preserving juice was canning. In canning, the juice is preserved from microbiological spoilage by rendering it commercially sterile and preventing its recontamination through a hermetic seal. Packaging for

canned juice can be made from metal or glass. The drawback to this method of juice preservation is a relatively rapid deterioration of the sensory properties of the juice. The juice begins to darken in color and develop "caramelized" or "cooked" flavors that are characteristic of flavors that develop during the cooking of foods. These changes in sensory quality are caused by so-called non-enzymatic browning and are generally considered undesirable in fruit juices.

Several approaches have been used to reduce or eliminate non-enzymatic browning in fruit juices, especially citrus juices, that are most sensitive to reduction in acceptability from these changes in sensory properties. The most popular approach to inhibit non-enzymatic browning in several varieties of fruit juices is to package the juices into cans that have an exposed metallic tin product contact surface. The acidic juice slowly dissolves some of the tin from the can's inner surface, thus preventing the formation of the dull color and "brown" taste characteristic of retorted juice stored in glass bottles. Of course this tin-leaching approach will not work for glass containers. This lead to the purposeful and quantified addition to the juice of tin ion in the form of stannous chloride. This approach has major drawbacks. The first drawback is that both methods produce a "canned" or metallic off taste. The second drawback is the addition of a metal to the juice that has no recognized dietary requirement.

Another additive approach to reduce or prevent non-enzymatic browning in fruit juices has been to add materials that produce sulfur dioxide when introduced into acidic juices. Sulfur dioxide may be added as a gas but is more conveniently added using one of the dry forms that are available in food grade forms. Sodium or potassium bisulfite or metabisulfite or simply sodium sulfite has all been demonstrated to inhibit enzymatic as well as non-enzymatic browning. Like the use of tin or its salts, this approach is partially effective in reducing color loss and somewhat less effective in reducing development of "brown" flavors. Also like tin or its salts, sulfur dioxide or its salts have major drawbacks. The most serious drawback to using sulfur dioxide is that it can function as an allergen and can pose certain levels of risk to people who have an allergic reaction to sulfur dioxide. The other drawback to using sulfur dioxide or its salts to inhibit non-enzymatic browning is the negative odor and flavor that is imparted by the additive.

Additives, both direct and indirect, have not been effective in eliminating the decline in sensory acceptability of fruit juice products during storage. Various investigators, convinced that this decline is due to non-enzymatic browning, have sought to remove or inactivate one or more of the reactants to stop development of the "brown" color and flavor. The primary action considered responsible for non-enzymatic browning of fruit juice is generally thought to be the Maillard reaction, which is responsible for the development of a purposeful brown color, similar to that which occurs when bread is baked to form a golden brown crust. While desirable for breads and meats these flavors and colors are not desirable in fruit juices. Maillard reactions occur between simple sugars and amines to form caramel, nutty, bready, or other "cooked" flavors and brown pigments. The sugars make up the major portion of the juice soluble solids while the amines are thought to be primarily derived from proteins in the juice. One of the major compounds resulting from these reactions is 5-hydroxymethyl-2-furaldehyde, commonly referred to as HMF. This non-enzymatic browning product is used as an indicator of thermal abuse in fruit juices.

The limited success and drawbacks of using additives have propelled investigators to develop other strategies to eliminate non-enzymatic browning in fruit juices. U.S. Patent No. 3,801,717 to Huffman describes a method using cation-exchange resins to remove amines, thus reducing the browning of fruit juices, particularly citrus juices, that are then dried into a powder form. This method, although partially successful in the preparation of dried juice, has not been demonstrated to benefit liquid juices or juice concentrates. The minimal amounts of amines remaining in the juice after treatment can initiate the production of off-flavors and dull colors. Thus, the treated juice is indistinguishable from the untreated juice. The method's success for dried juice applications is due to the loss of water. Water is an essential component for the Maillard reaction to proceed.

Removing the heat catalyst has been the only successful method to eliminate non-enzymatic browning in juices. This has been accomplished by freezing the juice or juice concentrate. During recent years pasteurized juices, also referred to as "not-from-concentrate products," have been held in a refrigerated state at temperatures just above the freezing point of the juice, but below the freezing point of

water. Even at these temperatures it is generally accepted that some deterioration of flavor occurs.

Frozen delivery and merchandising have obvious drawbacks of which include preventing consumers from consuming the juice until it has been thawed. Additionally, while thawing, packaging and distributing the juice using refrigerated distribution systems is highly developed in the USA, it is still unavailable at many popular occasions and locations. The refrigeration available in commercial food distribution is also at higher temperatures than is used for refrigerated juice inventories at the processing location. This few degrees' increase in temperature is enough to allow the non-enzymatic browning to proceed, thereby resulting in a shelf life limited to 50 to 70 days. Another drawback of inhibiting non-enzymatic browning by refrigeration is the lack of refrigerated distribution facilities at many popular venues.

Commercially produced fruit juice beverages are typically made with either concentrate or single strength juice. Beverages manufactured from juice concentrates are made by extracting fruit juice, concentrating the juice, reconstituting the juice, and adding flavorings and/or other additives to form a finished fruit juice which may then be packaged and stored. The juice may also be treated as needed to remove undesirable components, such as bitterness components, to make the finished juice beverage more palatable.

Certain taste and quality associated problems with current juice beverage systems are related to the manner in which the juices are packaged and stored. Many juices, especially citrus juices, need to be stored at refrigerated or reduced temperatures to prevent spoilage. Some of these juices will develop off-flavor or off-taste if they are stored at or above room temperature for extended periods of time. Accordingly, storage at refrigerated or reduced temperature helps to maintain taste and other sensory qualities. However, there are several disadvantages for this manner of storage. These refrigerated products require more complex shipping, handling, and retailing procedures. Also, consumers must store such juices in a refrigerated environment, even if they do not plan on using the juice immediately. Finally, refrigerated space is limited or non-existent in certain retail or consumption environments.

Spoilage problems have been addressed, in part, by the development of shelf-stable juices that retain acceptably low microbiological levels for a certain period of time. As used herein, "biologically shelf-stable" or "commercially sterile" refers to a juice that does not spoil due to microbial contamination when stored in non-refrigerated conditions for an extended period of time. However, these shelf-stable juices may suffer from off-flavor and/or off-taste in a short time period, for example, as little as one month or less under room-temperature storage.

In addition to off-taste and off-flavor that develops over time, certain fruit juices contain off-flavor components prior to storage. Certain processes have been developed for improving the taste of these fruit juices, such as grapefruit juices. One such process is directed to the removal of bitterness components from grapefruit juice, such as naringin, to improve the overall flavor of the juice. Bitterness removal is undertaken by passing the juice through a resin column containing a resin that selectively removes undesired bitterness components. However, these treated juices still require refrigeration to prevent the development of off-taste and/or off-flavor over time. Additionally, this process is only effective for juices that contain certain bitterness components. For example, grapefruit juice and naval orange juice, high in naringin and limonin, respectively, have been known to benefit from the debittering process. However, debittered grapefruit juices and naval orange juices are not intended for direct consumption, rather they are blended with other juices to produce juice beverages that require refrigeration to maintain product quality.

Certain juices have been found to have quality problems associated with taste, color, and other characteristics that exist for reasons other than the presence of such bitterness compounds. One such issue is the development of undesirable off-flavor and/or off-taste that occur in certain juices over time under non-refrigerated storage conditions.

Accordingly, there is a need for beverages, such as those containing fruit juices, having enhanced flavor and taste. Additionally, there is a need for beverages, such as fruit juices, capable of maintaining the flavor and taste of the beverage. Also, there is a need for a system for a beverage having desired quality aspects. Finally, there is a need for a fruit juice beverage having extended quality shelf-

life without the development of undesirable flavor and taste characteristics.

## SUMMARY OF THE INVENTION

Accordingly, it is an object of the present invention to provide fruit juices having enhanced quality including flavor and taste.

It is another object of the present invention to provide fruit juices having off-flavors and the precursors of off-flavors been removed and/or reduced to produce a fruit juice that maintains its flavor and taste quality for an extended period of time. Such extension of the shelf-life in quality-related characteristics such as flavor, taste, color, odor, mouthfeel, and other sensory characteristics shall be referred to as "quality shelf life" herein.

It is yet another object of the present invention to provide fruit juices having improved and extended quality shelf life, including flavor and taste characteristics.

It is yet another object of the present invention to provide fruit juice beverages having enhanced quality, flavor and taste.

It is yet another object of the present invention to provide fruit juice beverages having off-flavors and the precursors of off-flavors been removed or reduced to produce a beverage that maintains its flavor and taste quality for an extended period of time.

It is yet another object of the present invention to provide fruit juice beverages having improved and extended quality shelf life, including flavor and taste characteristics.

It is yet another object of the present invention to provide fruit juice beverages having improved and extended quality shelf life without nutritional loss.

Other objects, features, and advantages of the present invention will become apparent from the following description and claims.

The present invention fulfills the above-described objects by providing fruit juices having enhanced quality, including flavor and taste. In particular, the present invention is directed to fruit juices having enhanced quality including flavor and taste. This invention is especially useful with fruit juice beverages, such as citrus juice beverages. Additionally, the present invention is directed to fruit juices and fruit juice beverages having increased quality shelf-life. Finally, the

present invention is directed to fruit juices and fruit juice beverages having extended quality shelf-life without the development of off-taste or undesirable flavor or quality characteristics.

The present invention utilizes a system and method that is designed to remove off-flavors and/or the precursors of off-flavors from the fruit juice. By removing these off-flavors and/or the precursors of off-flavors, the present invention produces a finished fruit juice with quality characteristics, such as flavor and taste, that may be maintained for an extended time period. The off-flavors and the precursors of off-flavors are typically those which, if not removed, may increase in intensity, or degrade themselves, or interact with other compounds in the juice to form additional off-flavors and/or off-tastes over a period of time.

In certain fruit juices, quality-impacting off-flavors may be the result of off-flavor compounds present in the juice or may be formed from their precursors during storage, such as, for example, precursor compounds. Precursor compounds are present in small amounts in fresh juice. Furthermore, the sensory taste thresholds of these compounds are high and do not adversely affect the taste of fresh juice. However, over a period of time (about one month in the case of orange juice), the precursors will react with other components in the juices or degrade themselves to produce an undesirable off-taste and/or off-flavor in the juice if the juice is left in an non-refrigerated environment. The present invention, however, provides a fruit juice beverage with substantially reduced amounts of these off-flavors and/or precursors such that the juice will not only taste better after treatment, but will also maintain improved taste quality over an extended period of time, even if stored at room temperature.

The present invention may use any system or method that is capable of removing these off-flavor compounds and/or precursors. Different systems and methods may be used to remove these compounds to produce a finished juice product that has and maintains improved quality. The system or method may be used at any point in the production system for the finished juice product since the off-flavor compounds and precursors are present in the fresh juice product, and their removal does not adversely affect the remainder of the process by which the finished juice product is generated.

Therefore, by removing quality reducing off-flavors and precursors from certain juices, either freshly extracted or from the concentrate, the present invention includes a finished juice product with an extended quality shelf-life. If such a product is produced as a shelf stable product, then the juice is considered to be biologically shelf-stable at room temperature. A shelf stable product with an extended quality shelf life means a juice that may be stored at room temperature, i.e., from about 65° F to about 100° F, for a period in excess of one month, with a minimal increase in off-taste components, a corresponding minimal decrease in desirable quality attributes of flavor and taste, and without microbial spoilage. However, the present invention may also include those products that must be frozen or refrigerated to prevent microbial spoilage, as well as products which contain fruit juices, such as jams, jellies, or fruit preserves.

An added benefit of removing off-flavors and the precursors of off-flavors is that a fruit juice or fruit juice beverage having extended quality shelf-life may be produced. Not only will freshly treated juice maintain its taste for longer time periods, but by removing a substantial amount of the precursors, the juice may be stored at room temperatures for more than one month, without developing off-flavor and/or off-taste characteristics that have negatively impacted non-quality shelf-life juices of the prior art.

## DETAILED DESCRIPTION OF THE INVENTION

The present invention is directed to fruit juices having enhanced quality, flavor, and taste. These juices comprise a fruit juice that has been treated to remove off-flavor compounds and/or precursors to off-flavor compounds to reduced levels, depending on the off-flavor or precursor compound or compounds of interest. Once the off-flavor and/or precursor compound or compounds have been removed or substantially reduced, the treated juice will taste better. Additionally, the juice may be stored for extended periods of time without significant deterioration of the flavor and quality of the juice.

In fruit juices, it has been discovered that certain compounds affect the quality characteristics, such as flavors and/or tastes, of the fruit juice. Some of these compounds provide a bitter taste to the juice, while others simply mask the true flavor of the juice while producing an off-flavor or off-taste. Compounds that have been

identified as contributing to off-flavor development during ambient storage can be roughly classified into three categories: compounds derived from pasteurization/heat processing; compounds derived from non-enzymatic browning; and compounds derived from other degradations (i.e., Strecker degradation).

Typically, heated flavor is the most significant flavor attribute in the perception of quality and accounts for approximately 65% of the total variance with respect to the overall quality of orange juice. Traditionally, the thermally induced process flavors have been broadly viewed as products from browning reactions, because early discoveries showed the role of reducing sugars and amino compounds in the induction of a process that ultimately leads to the formation of brown pigments.

It is generally agreed that heat treatment of orange juice during pasteurization causes a heated juice flavor that increases in intensity as pasteurization conditions become more severe. Inactivation of enzymes requires even more heat treatment than pasteurization and thus contributes to flavor changes due to heat treatment. Sensory panels have been able to detect the heated flavor present in some heat-treated juice, especially those subjected to harsher time-temperature conditions. However, the components formed or changed immediately after heat treatment have been difficult to detect, and the few changes that have been documented were not studied for their contribution to heated juice flavor.

It has been reported that pasteurization for 1 second at 85°C (185°F) or 16 seconds at 74°C (165°F) are common adequate treatments for non-hermetic seal reconstituted juice from concentrate. Pasteurization at 91°C for 1 second was sufficient to reduce microorganisms to commercially sterile levels and to reduce enzyme (pectin methylesterase) activity by 97%. It is believed that certain compounds responsible for creating the heated/processed flavor begin to develop in the original concentrated orange juice, and only when the product is heated a second time do these compounds reach and/or exceed sensory threshold levels in the orange juice made from concentrate for certain individuals.

The second category of compounds that have been identified as attributing to off-flavor development during ambient storage are compounds derived from non-enzymatic browning.

Although browning reactions are almost always directly involved in the development of process flavors in foods, the interactions between degradation product of browning reaction and other food constitutes are also important and extensive to the development of other off-flavors.

Several compounds have been identified in canned single-strength orange juice after 12-weeks storage at 35°C. Most of the compounds are the degradation products of non-enzymatic browning and have impacted on the off-flavors developed during storage. These compounds may include off-flavors which grow in intensity over time or precursor compounds which eventually develop into off-flavors compounds over time.

Para-vinylguaiacol (PVG) is the most detrimental phenolic compound that contributes to an "old fruit" or "rotten" flavor in the juice. Levels of 0.075 ppm of PVG in freshly processed juice gives the juice an aged, off-flavor aroma, similar to that observed in stored juice. The formation of PVG from ferulic acid, a precursor to PVG, in orange juice has been demonstrated in a model system. The rate of PVG formation in orange juice during storage is time and temperature dependent. The limiting step in the formation of PVG in citrus products and the resulting objectionable odor, is perhaps the release of free ferulic acid from its bound forms (feruloylputrescine and feruloyllglucose) rather than the conversion of free ferulic acid to PVG. Pasteurization and an increase in mechanical pressure during fruit extraction will dramatically increase the release of ferulic acid from its bound forms. Due to its very low taste threshold (0.05 ppm) and its presence at levels above threshold found in freshly commercial orange juice, PVG is the major off-flavor in the early stage of thermally induced deterioration of citrus products.

The third category of compounds attributing to off-flavor development during ambient storage comprises compounds derived from other degradation such as Strecker degradation.

Strecker degradation involves the interaction of α-dicarbonyl compounds and α-amino acids. Volatile products, such as aldehydes, pyrazines, and sugar fragmentation products from Strecker reactions may contribute to aroma and flavor. Commercially, Strecker degradation is used to produce the distinctive flavors of chocolate, honey, maple syrup, and bread. Sulfur dioxide and sulfites have little

effect on Strecker degradation. Strecker degradation reaction transfers the amino group to the dicarbonyl group and provides a means from integrating amino acid nitrogen into small compounds destined for any other condensation mechanisms envisioned in these reactions. Strecker degradation, in which decarboxylation leads ultimately to colored products, is suggested by $CO_2$ formation, loss of reducing equivalents, and a decrease in ascorbate concentration.

The amount and potency of the off-flavor will increase over time, especially if the juice is stored in non-refrigerated conditions. The precursors of off-flavors, however, attribute to off-flavor development by degrading themselves or interacting with other components in the juice during ambient storage. Refrigeration helps to reduce the level of these off-flavor compounds by slowing down or stopping the reactions causing the increase of these off-flavor compounds. However, if the juice is stored at room temperature, these reactions will cause the juice to have such an offensive flavor or taste as to be considered undesirable and even undrinkable.

The present invention offers a unique solution to prior art methods by substantially removing the initial amounts of the off-flavor compounds and/or the precursors to these compounds. Once these compounds and/or precursors are removed or substantially reduced, the freshly treated fruit juice will taste better immediately after treatment and their removal will also allow the production of quality shelf-life fruit juices wherein the flavor, taste, color, odor, mouthfeel, and other sensory characteristics remain substantially unchanged. This preservation is achieved even if the juice is stored under non-refrigerated conditions for extended periods of time, such as one month or longer.

The present invention uses any system or method that is capable of removing or neutralizing these off-flavor compounds or the precursors of the off-flavor compounds. Example systems include, but are not limited to, resin systems, such as ion-exchange and adsorption resins; the use of treatment agents to prevent the degradation, such as a chelating and clarifying agents; additives, such as cysteine and its derivatives, antioxidants, or enzymes including genetic engineering and manipulation.

In a preferred embodiment, which is set forth in co-pending patent application U.S. Serial No. _____, the disclosure of

which is hereby incorporated by reference, the fruit juices of the present invention are formed by passing fruit juice through an adsorption resin selected to remove or substantially reduce the off-flavors and/or the precursors of off-flavors from the fruit juice. The resins used may be selected based upon the off-flavors or precursor compounds to be removed. For example, one class of resin compounds which may be used include those prepared by contacting a copolymer in a swollen state with a Friedel-Crafts catalyst under conditions effective to catalyze the post-crosslinking and rearrangement of the swollen copolymer. The total amount of compounds removed will vary, depending upon the types of juice treated and the initial level of the off-flavor compounds and/or the precursors.

The present invention may be used on any juice that includes off-flavor compounds and/or precursors to these compounds. The juices that may be treated include, but are not limited to, citrus juices such as orange juice, grapefruit juice, and tangerine juice; apple juice, pear juice, grape juice, pineapple juice, cranberry juice, and mixtures thereof, among others.

Once the off-flavor compounds and/or the precursors are substantially removed and/or reduced, the treated juice will immediately taste better, since the treatment lowers the level of the off-flavor compounds. The actual degree of removal differs among off-flavor compounds and the precursors of the off-flavors. Table 1 lists some of the markers and the desired level of these compounds in the treated juice. Those markers are found to be well correlated to the levels of the off-flavors and to the precursors of the off-flavors. Additionally, the levels for each compound are considered the upper desired limit. Preferably, the treated juices of the present invention have had all or substantially all of the off-flavors and/or precursors removed such that the lower limit for each compound is about 0 ppm. This lower level may vary depending on the treatment system used to remove the off-flavor compounds and/or the precursors. By treating the juice to reduce the off-flavors and/or precursors to the levels set forth in the Table 1, the treated juice and juice beverages will have an enhanced flavor quality for chilled orange juice (COJ) and extended quality shelf life for shelf stable orange juice. Different types of juice have different off-flavor components. A treated juice according to the present invention may be defined in terms of the level of a single marker or a

plurality of markers, depending on the treatment methods used and the juice being treated.

**Table 1. Markers And Their Desired Levels In A Treated Orange Juice According to the Present Invention**

| Marker | Preferred Level (ppm) | More Preferred Level (ppm) | Most Preferred Level (ppm) |
|---|---|---|---|
| Naringin | ≤ 70 | ≤ 55 | ≤ 40 |
| Narirutin Glycoside | ≤ 22 | ≤ 18 | ≤ 14 |
| Hesperidin Glycoside | ≤ 5.5 | ≤ 4.5 | ≤ 3.5 |

Additionally, since substantially all of the off-flavor compounds and/or precursors have been removed, the treated juice may be stored under non-refrigerated conditions without the development of off-flavor and/or off-tastes. It is preferred that the treated juice contains desired levels of the markers for off-flavor compounds and/or precursors as defined in Table 1. With these levels of the markers, the shelf-stable juices of the present invention will have an extended quality shelf life such that they will be able to be stored under non-refrigerated conditions for periods of time as long as six months, and maybe even longer, with little development of off-flavors or off-tastes. Table 2 lists some of the markers for the off-flavor compounds and/or precursors and their levels in treated juice that has been stored under non-refrigerated conditions. It is to be noted as well that since different types of juice have different off-flavor components and/or precursors, a treated juice according to the present invention may be defined in terms of the level of a single off-flavor component or precursor or a plurality of components and/or precursors, depending on the treatment methods used and the juice being treated.

**Table 2. Selected Markers and Their Levels in Treated Juice Stored at Non-Refrigerated Conditions**

| Weeks at 78°F | Samples | Naringin (ppm) | Naringin Glycoside (ppm) | Hesperidin Glycoside (ppm) |
|---|---|---|---|---|
| 0 | Control* | 83.8 | 35.2 | 9.5 |
| 0 | Treated | 35.9 | 12.9 | 3.7 |
| 3 | Control | 85.2 | 33.2 | 9.0 |
| 3 | Treated | 37.2 | 14.3 | 4.5 |
| 5 | Control | 83.3 | 36.5 | 10.9 |
| 5 | Treated | 36.8 | 13.2 | 4.4 |
| 10 | Control | 82.8 | 33.5 | 10.9 |
| 10 | Treated | 37.8 | 14.2 | 5.0 |
| 15 | Control | 83.5 | 31.1 | 12.5 |
| 15 | Treated | 37.6 | 13.1 | 5.6 |
| 20 | Control | 86.5 | 28.9 | 13.4 |
| 20 | Treated | 36.5 | 12.9 | 6.0 |

*Current hot-packed OJ with non-resin treated solids.

Another off-flavor component is PVG that increases dramatically during the ambient storage. Ferulic acid and its bound forms, feruloylputrescine and feruloyllglucose are known to be precursors to PVG. Accordingly, the present invention operates to a complete removal or substantial reduction of this off-flavor compound and/or its precursors as well if they are present in the fruit juice prior to treatment, thereby producing a treated fruit juice having reduced levels of the off-flavor compounds or precursors that develop into off-flavor (Table 3). As each fruit juice is different and the present invention includes a wide variety of juices, the actual levels of the different off-flavor or precursor compounds will vary with each type of treated juice. However, each desired treated juice will have maximum levels of those compounds which adversely affect the flavor of that type of juice the most.

**Table 3. Effect of Resin Treatment on PVG Formation in Shelf Stable Orange Juice Stored at Ambient Conditions*.**

| Sample | Week 0 | Week 5 | Week 10 | Week 15 | Week 20 |
|---|---|---|---|---|---|
| Control | 4.2 | 46.1 | 74.0 | 116.7 | 159.7 |
| Resin Treated | 0 | 29.9 | 46.1 | 72.8 | 92.3 |

*PVG concentration is expressed as parts per billion (ppb).

The present invention may also include, but is not limited to, means for removing solids, such as pulp, from the juice. These means could include a fixed strainer, ultrafiltration, a centrifuge or like means. Additionally, the invention may include means for supplying additives such as sweetening agents, flavor components, preservatives, nutritional or vitamin fortifications, pH adjusters, or mixtures thereof to the juice beverage. The invention may also include an evaporator for forming fruit juice concentrate, and means for adding water to reconstitute the juice. Finally, the invention may include microbial treatments such as pasteurization or other beverage manufacturing processes such as carbonation of the beverage, and the means for performing these functions.

The present invention yields a fruit juice that may be used as part of a beverage or other fruit juice product. These products include, but are not limited to, beverages, such as whole juices, blended juices, concentrated juices or juices used as ingredients in diluted juice beverages; jams; jellies; fruit preserves; or other foods. Accordingly, the juices may be post-treated in any manner necessary to produce the final juice product. For example, if the product is a beverage, the juice may have additional components added, such as sweeteners or flavor systems. Additionally, the beverage may be packaged in a desired manner, such as hot-packed or chill-packed, depending on the desired final product. Based on the reduced levels of off-flavors and/or precursors, the fruit juice beverages are shelf-stable and are able to be stored in non-refrigerated as well as refrigerated environments without the development of off-flavor or off-taste.

The present invention is further illustrated by the following examples, which are not to be construed in any way as imposing limitations upon the scope thereof. On the contrary, it is to be clearly understood that resort may be had to various other

embodiments, modifications, and equivalents thereof which, after reading the description herein, may suggest themselves to those skilled in the art without departing from the spirit of the present invention and/or the scope of the appended claims.

## EXAMPLES

### Example 1

To about 2 gallons of valencia orange juice (18° brix, 4% (v/v) fine-pulp), about 200 ml of the XAD-16 resin (Rohm and Haas Company, Philadelphia, PA) was added and gently stirred for one minute every 20 minutes. The resin was separated from the juice, one hour later, by 80 mesh sieve. The treated orange solids were then diluted to the final single strength (11.8° Brix) and flavor systems were added for packing. The hot-packed products were stored at 95°F and withdrawn weekly for evaluation by an expert panel for up to 3 weeks.

The results indicated that product with resin treated orange solids tasted significantly better than the controls. It had much less heated/cooked and off-oil flavors, which is true for samples withdrawn at 1, 2, and 3 weeks of storage at 95°F. The off-flavors developed in the control samples reached objectionable levels after one week of storage.

### Example 2

The orange solids with different fine pulp levels (0, 4, and 10%, v/v) were treated with the resin individually in a similar manner as described in Example 1. Non-resin-treated orange solids were used as the control. A total of 9 variables with about 3 gallons product of each were hot-packed and stored at 95°F for two weeks before being evaluated by the expert panel.

The results showed that the products with resin-treated orange solids had very little off-flavors while the control products tasted typically hot-packed and had strong off-flavors (cooked/heated notes and off oils). The difference between resin-treated and not resin-treated samples was much more significant than the difference between different pulp levels.

Examples 3-4

To confirm results from the laboratory studies and to explore the technical feasibility of commercialization, a scale-up pilot plant production was conducted. Orange concentrate blend (30 gallons, 65° brix) was diluted with hot water (90°F) to about 16° brix and passed through an ultra-filtration unit to removing the fine-pulp (retentate). The pulp-free orange serum (permeate) was then pumped through a commercial column filled with clean and equilibrated XAD-16 resin (7 gallons) at a rate of 2-3 gallons per minute. The resin treated orange permeate was blended with retentate and then concentrated to about 65 brix.

The resin-treated orange solids were used in two separate packing systems: 1) chilled orange juice; and 2) hot-packed shelf-stable orange juice. Both packing systems used non-resin treated orange solids (from same lot as resin-treated) as the control. The same flavor system was used for both the control and the resin-treated samples within the same product types. The products were tested by consumers at pre-selected time periods.

The results (Tables 4 & 5) demonstrated that even with the same flavor system, the product packed with resin treated orange solids obtained higher likability scores than their respective controls. The DA panel results confirmed consumer results that pre-treatment of orange solids with resin XAD-16 significantly improved the quality of both chilled and hot-packed orange juice when compared with the non-resin-treated controls.

**Table 4. Effect of Resin-Treatment on the Likability of Chilled Orange Juices***

| Samples | 3 weeks | 6 weeks | 8 weeks |
|---|---|---|---|
| Control | 6.3 | 6.4 | 6.4 |
| Resin-treated sample | 7.1 | 6.9 | 7.1 |

*Products were stored at 45°F. The likability is expressed as the hedonic scores in a 9-point scale with 1 as extremely dislike and 9 as extremely like. Scores in the 3 and 8 week columns are significantly different at 5% confidence level while scores in the 6-week column is significantly different at 10% confidence level.

**Table 5. Effect of Resin-Treatment on the Likability of Shelf Stable Orange Juices***

| Samples | 5 weeks | 10 weeks | 15 weeks |
|---|---|---|---|
| Control | 6.2 | 5.7 | 5.5 |
| Resin-treated sample | 6.9 | 6.5 | 6.1 |

*Products were stored at 78°F. The likability is expressed as the hedonic scores in a 9-point scale with 1 as extremely dislike and 9 as extremely like. All scores in the same column are significantly different at 5% confidence level.

Example 5

An independent consumer study was run in parallel to the study described in Example 4. This study involved two three-sample likability tests conducted at 8 and 13 weeks of ambient storage, respectively. The prototypes were formulated with resin-treated solids and different Minute Maid® flavor systems, and the control was a current commercial shelf-stable orange juice. The consumer test results indicated that the two prototypes were significantly ($p \leq 0.05$) liked by consumers more than was the control during these periods (Table 6).

**Table 6. Effect of Resin-Treatment and Flavor System on the Likability of Shelf Stable Orange Juices***

| Samples | 8 weeks | 13 weeks |
|---|---|---|
| Control | 5.4B | 5.0b |
| Prototype 1 | 6.6A | 6.3a |
| Prototype 2 | 6.2A | 6.2a |

*Means preceded by the same letter are not significantly ($p \leq 0.05$) different in the same column.

Example 6

Previous consumer tests indicated that concentrated orange juice ("COJ") with resin treated solids was significantly better liked than the current COJ during 3-8 weeks of refrigerated storage (45°F). To validate these results, a large-scale consumer test was conducted in 4 different locations around the United States with a total of 150 consumers. A bulk panel carefully screened orange concentrate so that the concentrate used for the product would be free of defects and bitterness. A two-sample likability test (control vs. treated COJ)

was conducted with a forced preference test at the end. The results confirmed our previous findings.

At week 3, the COJ with resin-treated solids was significantly ($p \leq 0.05$) better liked and more preferred than the control. At week 7, the product with resin-treated solids was directionally liked and significantly more preferred ($p \leq 0.05$) than the control.

Example 7

To show the improvement provided by the juices of the present invention, a brief survey was conducted on selected commercial shelf-stable orange juices sold in the local market. As shown in Table 6, all the markers in the commercial shelf-stable orange juice purchased from the local market are well above the desired levels of off-flavors for the juices of the present invention, indicating that treated juices taste better and have longer shelf life when compared to non-treated juices.

**Table 6. Survey on Selected Commercial Shelf-Stable Orange Juice***

| Sample (Brand Name) | Naringin (ppm) | Naringin Glycoside (ppm) | Hesperidin Glycoside (ppm) | PVG (ppb) |
|---|---|---|---|---|
| Tropicana® Season's Best | 78.7 | 26.7 | 7.7 | 21.0 |
| Minute Maid® | 78.3 | 29.2 | 11.5 | 48.2 |
| Ocean Spray® | 78.5 | 29.1 | 11.3 | 115.2 |
| Hohes C® | 81.3 | 28.1 | 9.9 | 53.4 |
| Velda Farms® | 90.8 | 28.2 | 5.8 | 53.0 |
| Treated Juice | ≤ 70 | ≤ 22 | ≤ 5.5 | ** |

*All the data are expressed as the means of 2-4 replicated measurements.
**Will vary depending on the conditions and the length of time the juice is stored. Desired level immediately after treatment is ≤ 4.0 ppb.

Thus, the present invention provides fruit juices having enhanced flavor and overall quality and provides a shelf-stable fruit juices with an extended quality shelf life. The juices may be prepared for sale as whole juices, blended juices, concentrated juices or juices used as ingredients in diluted juice beverages, jams, jellies, fruit preserves or other foods that may deteriorate due to quality deterioration of fruit juice.

# CLAIMS

We claim:

1. A fruit juice comprising a treated fruit juice containing less than about 70 ppm of Narirutin.

2. The fruit juice of Claim 1, wherein the treated fruit juice contains less than about 55 ppm Narirutin.

3. The fruit juice of Claim 2, wherein the treated fruit juice contains less than about 40 ppm Narirutin.

4. The fruit juice of Claim 1, wherein the treated fruit juice further contains less than about 22 ppm of Narirutin Glycoside.

5. The fruit juice of Claim 2, wherein the treated fruit juice further contains less than about 22 ppm Narirutin Glycoside.

6. The fruit juice of Claim 3, wherein the treated fruit juice further contains less than about 22 ppm Narirutin Glycoside.

7. The fruit juice of Claim 1, wherein the treated fruit juice further contains less than about 5.5 ppm of Hesperidin Glycoside.

8. The fruit juice of Claim 2, wherein the treated fruit juice further contains less than about 5.5 ppm of Hesperidin Glycoside.

9. The fruit juice of Claim 3, wherein the treated fruit juice further contains less than about 5.5 ppm of Hesperidin Glycoside.

10. The fruit juice of Claim 1, wherein the treated fruit juice further contains less than about 4.0 ppb para-vinylguaiacol.

11. The fruit juice of Claim 1, wherein the fruit juice is selected from orange juice, grapefruit juice, and tangerine juice; apple juice, pear juice, grape juice, pineapple juice, cranberry juice, or mixtures thereof.

12. A fruit juice product comprising the fruit juice of Claim 1, the fruit juice product selected from the group consisting of whole juices; blended juices; concentrated juices; juices used as ingredients in diluted juice beverages; jams; jellies; fruit preserves; or other foods.

13. The fruit juice product of Claim 12, wherein the fruit juice product can be stored under storage conditions selected from the group consisting of ambient, refrigerated, or frozen conditions.

14. A fruit juice comprising a treated fruit juice containing less than about 22 ppm of Narirutin Glycoside.

15. The fruit juice of Claim 14, wherein the treated fruit juice contains less than about 18 ppm Narirutin Glycoside.

16. The fruit juice of Claim 15, wherein the treated fruit juice contains less than about 14 ppm Narirutin Glycoside.

17. The fruit juice of Claim 15, wherein the treated fruit juice further contains less than about 70 ppm of Narirutin.

18. The fruit juice of Claim 16, wherein the treated fruit juice further contains less than about 70 ppm Narirutin.

19. The fruit juice of Claim 15, wherein the treated fruit juice further contains less than about 55 ppm of Narirutin.

20. The fruit juice of Claim 16, wherein the treated fruit juice further contains less than about 55 ppm Narirutin.

21. The fruit juice of Claim 15, wherein the treated fruit juice further contains less than about 40 ppm of Narirutin.

22. The fruit juice of Claim 16, wherein the treated fruit juice further contains less than about 40 ppm Narirutin.

23. The fruit juice of Claim 14, wherein the treated fruit juice further contains less than about 5.5 ppm of Hesperidin Glycoside.

24. The fruit juice of Claim 15, wherein the treated fruit juice further contains less than about 5.5 ppm of Hesperidin Glycoside.

25. The fruit juice of Claim 16, wherein the treated fruit juice further contains less than about 5.5 ppm of Hesperidin Glycoside.

26. The fruit juice of Claim 14, wherein the treated fruit juice further contains less than about 4.0 ppb para-vinylguaiacol.

27. The fruit juice of Claim 14, wherein the fruit juice is selected from orange juice, grapefruit juice, and tangerine juice; apple juice, pear juice, grape juice, pineapple juice, cranberry juice, or mixtures thereof.

28. A fruit juice product comprising the fruit juice of Claim 14, the fruit juice product selected from the group consisting of whole juices; blended juices; concentrated juices; juices used as ingredients in diluted juice beverages; jams; jellies; fruit preserves; or other foods.

29. The fruit juice product of Claim 28, wherein the fruit juice product can be stored under storage conditions selected from the group consisting of ambient, refrigerated, or frozen conditions.

30. A fruit juice comprising a treated fruit juice containing less than about 5.5 ppm of Hesperidin Glycoside.

31. The fruit juice of Claim 30, wherein the treated fruit juice contains less than about 4.5 ppm Hesperidin Glycoside.

32. The fruit juice of Claim 31, wherein the treated fruit juice contains less than about 3.5 ppm Hesperidin Glycoside.

33. The fruit juice of Claim 31, wherein the treated fruit juice further contains less than about 70 ppm of Narirutin.

34. The fruit juice of Claim 32, wherein the treated fruit juice further contains less than about 70 ppm Narirutin.

35. The fruit juice of Claim 31, wherein the treated fruit juice further contains less than about 55 ppm of Narirutin.

36. The fruit juice of Claim 32, wherein the treated fruit juice further contains less than about 55 ppm Narirutin.

37. The fruit juice of Claim 31, wherein the treated fruit juice further contains less than about 40 ppm of Narirutin.

38. The fruit juice of Claim 32, wherein the treated fruit juice further contains less than about 40 ppm Narirutin.

39. The fruit juice of Claim 31, wherein the treated fruit juice further contains less than about 22 ppm of Narirutin Glycoside.

40. The fruit juice of Claim 32, wherein the treated fruit juice further contains less than about 22 ppm Narirutin Glycoside.

41. The fruit juice of Claim 31, wherein the treated fruit juice further contains less than about 18 ppm of Narirutin Glycoside.

42. The fruit juice of Claim 32, wherein the treated fruit juice further contains less than about 18 ppm Narirutin Glycoside.

43. The fruit juice of Claim 31, wherein the treated fruit juice further contains less than about 14 ppm of Narirutin Glycoside.

44. The fruit juice of Claim 32, wherein the treated fruit juice further contains less than about 14 ppm Narirutin Glycoside.

45. The fruit juice of Claim 30, wherein the treated fruit juice further contains less than about 4.0 ppb para-vinylguaiacol.

46. The fruit juice of Claim 30, wherein the fruit juice is selected from orange juice, grapefruit juice, and tangerine juice; apple

juice, pear juice, grape juice, pineapple juice, cranberry juice, or mixtures thereof.

47. A fruit juice product comprising the fruit juice of Claim 30, the fruit juice product selected from the group consisting of whole juices; blended juices; concentrated juices; juices used as ingredients in diluted juice beverages; jams; jellies; fruit preserves; or other foods.

48. The fruit juice product of Claim 47, wherein the fruit juice product can be stored under storage conditions selected from the group consisting of ambient, refrigerated, or frozen conditions.

49. A fruit juice comprising a treated fruit juice containing less than about 70 ppm of Narirutin, less than about 22 ppm of Narirutin Glycoside, and less than about 5.5 ppm of Hesperidin Glycoside.

50. The fruit juice of Claim 49, wherein the treated fruit juice further contains less than about 4.0 ppb para-vinylguaiacol.

51. The fruit juice of Claim 49, wherein the fruit juice is selected from orange juice, grapefruit juice, and tangerine juice; apple juice, pear juice, grape juice, pineapple juice, cranberry juice, or mixtures thereof.

52. A fruit juice product comprising the fruit juice of Claim 49, the fruit juice product selected from the group consisting of whole juices; blended juices; concentrated juices; juices used as ingredients in diluted juice beverages; jams; jellies; fruit preserves; or other foods.

53. The fruit juice product of Claim 52, wherein the fruit juice product can be stored under storage conditions selected from the group consisting of ambient, refrigerated, or frozen conditions.

54. A shelf-stable fruit juice comprising a treated fruit juice containing less than about 4.0 ppb para-vinylguaiacol.

55. The shelf-stable fruit juice of Claim 54, wherein, after storage from about 65°F to about 100°F for about 5 weeks, the treated fruit juice contains less than about 35 ppb para-vinylguaiacol.

56. The shelf-stable fruit juice of Claim 54, wherein, after storage from about 65°F to about 100°F for about 10 weeks, the treated fruit juice contains less than about 60 ppb para-vinylguaiacol.

57. The shelf-stable fruit juice of Claim 54, wherein, after storage from about 65°F to about 100°F for about 15 weeks, the treated fruit juice contains less than about 100 ppb para-vinylguaiacol.

58. The shelf-stable fruit juice of Claim 54, wherein, after storage from about 65°F to about 100°F for about 20 weeks, the treated fruit juice contains less than about 140 ppb para-vinylguaiacol.

59. The shelf-stable fruit juice of Claim 54, wherein the treated fruit juice contains less than about 70 ppm of Narirutin.

60. The shelf-stable fruit juice of Claim 54, wherein the treated fruit juice contains less than about 22 ppm of Narirutin Glycoside.

61. The shelf-stable fruit juice of Claim 54, wherein the treated fruit juice contains less than about 5.5 ppm of Hesperidin Glycoside.

62. The shelf-stable fruit juice of Claim 54, wherein the treated fruit juice contains less than about 70 ppm of Narirutin, less than about 22 ppm of Narirutin Glycoside, and less than about 5.5 ppm of Hesperidin Glycoside.

63. The fruit juice of Claim 54, wherein the fruit juice is selected from orange juice, grapefruit juice, and tangerine juice; apple juice, pear juice, grape juice, pineapple juice, cranberry juice, or mixtures thereof.

64. A fruit juice product comprising the fruit juice of Claim 1, the fruit juice product selected from the group consisting of whole juices; blended juices; concentrated juices; juices used as ingredients in diluted juice beverages; jams; jellies; fruit preserves; or other foods.